Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(Dibenzo-18-crown-6)(2-phenylamidopyridine)-potassium(I)

William Clegg* and Stephen T. Liddle‡

School of Natural Sciences (Chemistry), University of Newcastle upon Tyne, Newcastle upon Tyne NE1 7RU, England

‡ Current address: School of Chemistry, University of Nottingham, University Park, Nottingham NG7 2RD, England

Correspondence e-mail: w.clegg@ncl.ac.uk

Key indicators

Single-crystal X-ray study
T = 160 K
Mean σ(C–C) = 0.003 Å
R factor = 0.043
wR factor = 0.110
Data-to-parameter ratio = 18.5

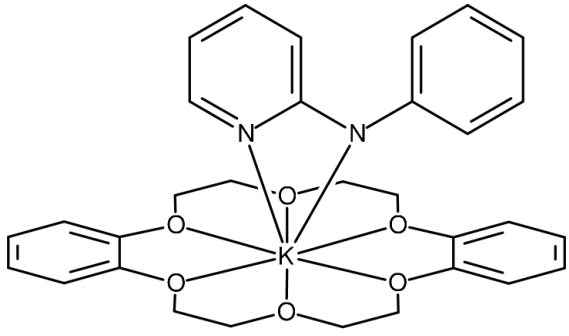
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 14 September 2004
Accepted 20 September 2004
Online 30 September 2004

The title complex, $[Rb(C_{11}H_9N_2)(C_{20}H_{24}O_6)]$, has potassium in an irregular eightfold coordination, with a hexadentate crown ether ligand and a chelating bidentate amidopyridine ligand, each occupying one hemisphere of the coordination. The chelate KNCN ring is slightly folded, and the two rings of the amidopyridine ligand are not coplanar, because of steric interaction of H atoms on the rings. The K—N(amido) bond is shorter than the K—N(pyridine) bond. The coordination of the crown ether to potassium is less symmetrical than that for the analogous rubidium complex, reflecting a poorer size match for K^+ in this coordination site.

Comment

In the preceding paper (Liddle & Clegg, 2004), we described the structure of the complex (dibenzo-18-crown-6)Rb(*L*), where H*L* is 2-phenylaminopyridine. We present here the structure of the analogous potassium complex, (dibenzo-18-crown-6)K(*L*), (I). These complexes were prepared as part of a study of crown-ether-supported complexes of alkali metals with amide ligands, and were investigated specifically for comparison with the complexes with 18-crown-6 (Liddle *et al.*, 2004; Liddle & Clegg, 2003).



(I)

Although the Rb complexes with the two different crown ethers are structurally very similar, there is a marked difference for the K complexes. Reaction of equimolar amounts of 18-crown-6, potassium hydride and H*L* leads to the polymeric complex $[(18\text{-crown-6})K(L)_2K]_\infty$, even though this product has a 1:2:2 stoichiometry (Liddle *et al.*, 2004). With dibenzo-18-crown-6 instead of 18-crown-6, the title complex (with a 1:1:1 stoichiometry) is obtained, exactly analogous to the Rb case.

The dibenzo-18-crown-6 complexes of Rb and K are structurally similar in gross terms; there are subtle but significant differences in detail. Fig. 1 shows the molecular structure of the title K complex, and selected geometric

parameters are in Table 1. It consists of discrete neutral molecules with no special intermolecular interactions. The hexadentate crown ligand and the bidentate amide anion occupy the two coordination hemispheres of the potassium ion, giving irregular eightfold coordination, and the complex may be described as a contact ion pair.

The range of K—O distances and the difference between the two K—N distances are both somewhat greater than the corresponding values for the Rb complex, and this probably reflects the poorer fit of the smaller potassium ion in the coordination site; it is generally recognized that 18-crown-6 and its substituted derivatives provide an ideal fit for K^+ in the mean plane of the six O atoms rather than displaced from this plane. In the title complex, K lies 0.7830 (7) Å out of the oxygen mean plane (r.m.s. deviation 0.009 Å), compared with a deviation of 1.0945 (6) Å for Rb in the analogous complex. Although all four O—C—C—O aliphatic segments have a *gauche* conformation, as is expected for optimal chelation, the overall conformation of the crown in this complex differs from that in the Rb complex by conversion of one *anti* C—C—O—C linkage to *gauche*, the other eleven remaining *anti* (Table 1). The crown ligand is thus rather less symmetrical in the title complex than in the Rb complex. The two benzene rings are folded out of the oxygen mean plane, away from the amide ligand by 39.97 (6) and 8.52 (7)°, in contrast to the angles of 22.93 (7) and 25.94 (7)° towards the amide ligand in the Rb complex.

The two rings of the amide ligand have a dihedral angle of 49.91 (7)° because of steric interaction of H atoms bonded to C4 and C11. The four-membered chelate ring (KNCN) is approximately planar, the dihedral angle between the KN_2 and CN_2 planes being 7.3 (2)°, compared with only 0.3 (3)° in the Rb complex, once again displaying the effects of greater geometrical strain from the size mismatch of the metal ion and its ligand set.

Experimental

Potassium hydride (0.04 g, 1.0 mmol) was added to a solution of 2-phenylaminopyridine (0.17 g, 1.0 mmol) and dibenzo-18-crown-6 (0.36 g, 1.0 mmol) in tetrahydrofuran (THF, 40 ml), to give a pale yellow precipitate. Volatile components were removed *in vacuo* and the remaining solid was washed with petroleum ether (3 × 5 ml). Recrystallization from hot toluene containing a little hexamethylphosphoramide (HMPA) gave yellow crystals of (I) (yield 0.38 g, 67%). Chemical analysis results were satisfactory, and the ^{1}H and ^{13}C{^{1}H} NMR signals could be assigned on the basis of the crystal structure (Liddle, 2000).

Crystal data

$[K(C_{11}H_9N_2)(C_{20}H_{24}O_6)]$
M_r = 568.69
Monoclinic, $C2/c$
a = 26.950 (2) Å
b = 10.3120 (9) Å
c = 22.8660 (19) Å
β = 117.607 (2)°
V = 5631.0 (8) Å^3
Z = 8
D_x = 1.342 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7626 reflections
θ = 2.5–27.8°
μ = 0.24 mm^{-1}
T = 160 (2) K
Needle, yellow
0.82 × 0.12 × 0.10 mm

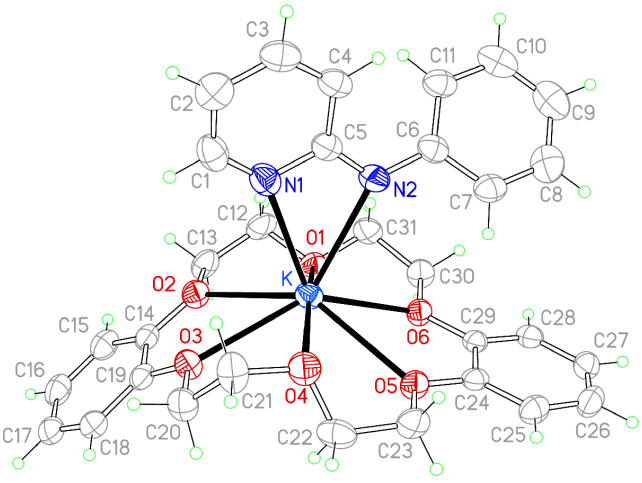


Figure 1
The molecular structure of (I), showing the atom labels and 50% probability displacement ellipsoids for non-H atoms.

Data collection

Bruker SMART 1K CCD diffractometer
Thin-slice ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.830, T_{max} = 0.977
21735 measured reflections
6673 independent reflections
4349 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 28.6°
$h = -35 \rightarrow 35$
$k = -13 \rightarrow 12$
$l = -30 \rightarrow 29$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.110
S = 1.04
6673 reflections
361 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0525P)^2 + 1.0053P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.77 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

K—N1	2.8515 (18)	K—O5	2.8374 (12)
K—N2	2.7935 (16)	K—O6	2.8194 (12)
K—O1	2.8156 (13)	N1—C1	1.331 (3)
K—O2	2.8578 (12)	N1—C5	1.368 (2)
K—O3	2.8519 (12)	N2—C5	1.340 (2)
K—O4	2.7138 (13)	N2—C6	1.384 (2)
N1—K—N2	47.72 (5)	K—N1—C1	143.34 (14)
O1—K—O2	60.36 (4)	K—N1—C5	96.70 (12)
O1—K—O6	58.97 (3)	C1—N1—C5	118.40 (18)
O2—K—O3	54.13 (3)	K—N2—C5	100.10 (11)
O3—K—O4	61.85 (4)	K—N2—C6	136.59 (12)
O4—K—O5	60.48 (4)	C5—N2—C6	123.11 (16)
O5—K—O6	54.89 (3)	N1—C5—N2	115.03 (17)
C6—N2—C5—N1	168.74 (17)	C22—O4—C21—C20	−67.4 (2)
C6—N2—C5—C4	−16.0 (3)	O3—C20—C21—O4	−58.0 (2)
C5—N2—C6—C7	145.31 (18)	C21—O4—C22—C23	−170.92 (14)
C5—N2—C6—C11	−40.6 (3)	C24—O5—C23—C22	−171.65 (15)
C31—O1—C12—C13	−179.51 (15)	O4—C22—C23—O5	−62.1 (2)
C14—O2—C13—C12	−174.33 (15)	C23—O5—C24—C29	−175.53 (16)
O1—C12—C13—O2	68.2 (2)	C30—O6—C29—C24	175.55 (16)
C13—O2—C14—C19	170.87 (15)	O5—C24—C29—O6	3.8 (2)
C20—O3—C19—C14	175.63 (15)	C29—O6—C30—C31	−158.91 (15)
O2—C14—C19—O3	2.3 (2)	C12—O1—C31—C30	−179.30 (15)
C19—O3—C20—C21	−168.71 (16)	O6—C30—C31—O1	−59.1 (2)

H atoms were positioned geometrically, with C—H = 0.95 (aromatic) or 0.99 Å (aliphatic), and refined with a riding model, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 2001); cell refinement: local programs; data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and local programs.

We thank the EPSRC for financial support.

References

Bruker (2001). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Liddle, S. T. (2000). PhD thesis, University of Newcastle upon Tyne, England.
Liddle, S. T. & Clegg, W. (2003). *Polyhedron*, **22**, 3507–3513.
Liddle, S. T. & Clegg, W. (2004). *Acta Cryst.* E**60**, m1492–m1494.
Liddle, S. T., Clegg, W. & Morrison, C. A. (2004). *Dalton Trans.* pp. 2514–2525.
Sheldrick, G. M. (2001). *SHELXTL*. Version 5. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2002). *SADABS*. University of Göttingen, Germany.

supporting information

Acta Cryst. (2004). E**60**, m1495–m1497 [https://doi.org/10.1107/S1600536804023360]

(Dibenzo-18-crown-6)(2-phenylamidopyridine)potassium(I)

William Clegg and Stephen T. Liddle

(Dibenzo-18-crown-6)(2-phenylamidopyridine)potassium(I)

Crystal data

$[K(C_{11}H_9N_2)(C_{20}H_{24}O_6)]$
$M_r = 568.69$
Monoclinic, $C2/c$
$a = 26.950$ (2) Å
$b = 10.3120$ (9) Å
$c = 22.8660$ (19) Å
$\beta = 117.607$ (2)°
$V = 5631.0$ (8) Å^3
$Z = 8$
$F(000) = 2400$
$D_x = 1.342$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7626 reflections
$\theta = 2.5$–27.8°
$\mu = 0.24$ mm^{-1}
$T = 160$ K
Needle, yellow
0.82 × 0.12 × 0.10 mm

Data collection

Bruker SMART 1K CCD diffractometer
Radiation source: sealed tube
Graphite monochromator
Detector resolution: 8.192 pixels mm^{-1}
thin–slice ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2002)
$T_{min} = 0.830$, $T_{max} = 0.977$
21735 measured reflections
6673 independent reflections
4349 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 28.6°$, $\theta_{min} = 1.7°$
$h = -35 \rightarrow 35$
$k = -13 \rightarrow 12$
$l = -30 \rightarrow 29$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.110$
$S = 1.04$
6673 reflections
361 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0525P)^2 + 1.0053P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.77$ e Å^{-3}
$\Delta\rho_{min} = -0.35$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
K	0.147063 (16)	0.53950 (4)	0.039410 (19)	0.03171 (12)
N1	0.10174 (7)	0.75945 (17)	−0.04426 (8)	0.0419 (4)
N2	0.10649 (7)	0.77716 (16)	0.05777 (8)	0.0403 (4)

C1	0.08226 (8)	0.8005 (2)	−0.10619 (10)	0.0439 (5)
H1A	0.0922	0.7509	−0.1342	0.053*
C2	0.04928 (9)	0.9074 (2)	−0.13350 (11)	0.0484 (5)
H2A	0.0370	0.9316	−0.1782	0.058*
C3	0.03478 (8)	0.9785 (2)	−0.09186 (10)	0.0440 (5)
H3A	0.0111	1.0520	−0.1083	0.053*
C4	0.05458 (8)	0.94262 (19)	−0.02746 (10)	0.0371 (5)
H4A	0.0457	0.9935	0.0011	0.045*
C5	0.08864 (7)	0.82902 (19)	−0.00237 (10)	0.0338 (4)
C6	0.10561 (7)	0.84335 (19)	0.10993 (9)	0.0359 (4)
C7	0.09463 (8)	0.7730 (2)	0.15545 (10)	0.0396 (5)
H7A	0.0857	0.6834	0.1477	0.048*
C8	0.09632 (9)	0.8296 (2)	0.21069 (10)	0.0507 (6)
H8A	0.0882	0.7790	0.2399	0.061*
C9	0.10969 (9)	0.9589 (3)	0.22430 (11)	0.0555 (6)
H9A	0.1106	0.9980	0.2624	0.067*
C10	0.12162 (8)	1.0302 (2)	0.18134 (11)	0.0498 (6)
H10A	0.1314	1.1191	0.1904	0.060*
C11	0.11964 (8)	0.9746 (2)	0.12542 (11)	0.0421 (5)
H11A	0.1279	1.0263	0.0967	0.051*
O1	0.25742 (5)	0.59682 (12)	0.13446 (6)	0.0334 (3)
O2	0.23595 (5)	0.52324 (13)	0.00460 (6)	0.0345 (3)
O3	0.14500 (5)	0.39465 (12)	−0.06794 (6)	0.0329 (3)
O4	0.05117 (5)	0.41353 (14)	−0.04173 (6)	0.0413 (3)
O5	0.09313 (5)	0.38149 (12)	0.09450 (6)	0.0330 (3)
O6	0.18057 (5)	0.51384 (12)	0.17537 (6)	0.0328 (3)
C12	0.27351 (9)	0.6723 (2)	0.09390 (10)	0.0421 (5)
H12A	0.3068	0.7252	0.1218	0.051*
H12B	0.2427	0.7317	0.0661	0.051*
C13	0.28664 (8)	0.5838 (2)	0.05116 (9)	0.0398 (5)
H13A	0.3033	0.6337	0.0277	0.048*
H13B	0.3139	0.5171	0.0786	0.048*
C14	0.24077 (8)	0.42932 (18)	−0.03513 (9)	0.0311 (4)
C15	0.28920 (8)	0.4022 (2)	−0.03978 (10)	0.0409 (5)
H15A	0.3222	0.4512	−0.0148	0.049*
C16	0.28944 (9)	0.3031 (2)	−0.08100 (10)	0.0432 (5)
H16A	0.3228	0.2848	−0.0839	0.052*
C17	0.24219 (9)	0.2317 (2)	−0.11733 (10)	0.0409 (5)
H17A	0.2428	0.1635	−0.1449	0.049*
C18	0.19340 (8)	0.25955 (18)	−0.11352 (9)	0.0360 (4)
H18A	0.1605	0.2104	−0.1388	0.043*
C19	0.19227 (7)	0.35806 (18)	−0.07329 (8)	0.0301 (4)
C20	0.09366 (8)	0.3299 (2)	−0.10991 (9)	0.0391 (5)
H20A	0.0949	0.2391	−0.0952	0.047*
H20B	0.0879	0.3290	−0.1559	0.047*
C21	0.04672 (8)	0.4021 (2)	−0.10602 (9)	0.0440 (5)
H21A	0.0447	0.4904	−0.1240	0.053*
H21B	0.0111	0.3578	−0.1348	0.053*

C22	0.04578 (9)	0.2961 (2)	−0.01491 (9)	0.0436 (5)
H22A	0.0786	0.2404	−0.0053	0.052*
H22B	0.0118	0.2502	−0.0471	0.052*
C23	0.04172 (8)	0.3205 (2)	0.04790 (9)	0.0379 (5)
H23A	0.0095	0.3778	0.0389	0.046*
H23B	0.0363	0.2377	0.0661	0.046*
C24	0.10019 (7)	0.39557 (17)	0.15784 (9)	0.0285 (4)
C25	0.06357 (8)	0.34800 (19)	0.17935 (9)	0.0359 (4)
H25A	0.0306	0.3037	0.1495	0.043*
C26	0.07504 (8)	0.3650 (2)	0.24480 (10)	0.0413 (5)
H26A	0.0496	0.3328	0.2593	0.050*
C27	0.12269 (8)	0.42753 (19)	0.28840 (10)	0.0381 (5)
H27A	0.1304	0.4374	0.3331	0.046*
C28	0.15986 (8)	0.47670 (18)	0.26747 (9)	0.0325 (4)
H28A	0.1931	0.5193	0.2980	0.039*
C29	0.14839 (7)	0.46343 (17)	0.20197 (9)	0.0279 (4)
C30	0.22798 (8)	0.5921 (2)	0.21820 (9)	0.0378 (5)
H30A	0.2598	0.5357	0.2466	0.045*
H30B	0.2181	0.6465	0.2470	0.045*
C31	0.24386 (8)	0.67620 (19)	0.17617 (9)	0.0378 (5)
H31A	0.2123	0.7344	0.1489	0.045*
H31B	0.2765	0.7305	0.2046	0.045*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K	0.0283 (2)	0.0301 (2)	0.0363 (2)	−0.00319 (17)	0.01459 (17)	−0.00488 (18)
N1	0.0407 (10)	0.0413 (10)	0.0480 (10)	0.0019 (8)	0.0241 (9)	−0.0050 (8)
N2	0.0455 (10)	0.0303 (10)	0.0514 (10)	0.0059 (7)	0.0279 (9)	0.0009 (8)
C1	0.0385 (11)	0.0501 (14)	0.0480 (13)	−0.0072 (10)	0.0243 (10)	−0.0132 (11)
C2	0.0422 (12)	0.0519 (14)	0.0437 (12)	−0.0096 (11)	0.0135 (10)	−0.0036 (11)
C3	0.0354 (11)	0.0343 (12)	0.0539 (13)	−0.0046 (9)	0.0135 (10)	−0.0034 (10)
C4	0.0305 (10)	0.0306 (11)	0.0504 (12)	−0.0021 (8)	0.0188 (9)	−0.0054 (9)
C5	0.0262 (9)	0.0307 (11)	0.0483 (12)	−0.0057 (8)	0.0204 (9)	−0.0050 (9)
C6	0.0282 (10)	0.0346 (11)	0.0429 (11)	0.0063 (8)	0.0148 (9)	−0.0002 (9)
C7	0.0377 (11)	0.0347 (12)	0.0436 (12)	0.0062 (9)	0.0164 (9)	0.0066 (9)
C8	0.0455 (12)	0.0655 (17)	0.0328 (11)	0.0113 (11)	0.0111 (10)	0.0089 (11)
C9	0.0502 (14)	0.0667 (18)	0.0345 (12)	0.0122 (12)	0.0068 (10)	−0.0092 (12)
C10	0.0359 (11)	0.0429 (13)	0.0528 (13)	0.0041 (10)	0.0055 (10)	−0.0121 (11)
C11	0.0356 (11)	0.0341 (12)	0.0557 (13)	−0.0012 (9)	0.0203 (10)	−0.0023 (10)
O1	0.0344 (7)	0.0303 (7)	0.0372 (7)	−0.0076 (5)	0.0181 (6)	−0.0014 (6)
O2	0.0305 (7)	0.0371 (8)	0.0377 (7)	−0.0063 (6)	0.0172 (6)	−0.0032 (6)
O3	0.0281 (7)	0.0366 (8)	0.0343 (7)	−0.0029 (5)	0.0148 (6)	−0.0058 (6)
O4	0.0387 (8)	0.0444 (9)	0.0412 (8)	−0.0078 (6)	0.0188 (6)	−0.0066 (6)
O5	0.0297 (7)	0.0365 (8)	0.0325 (7)	−0.0114 (5)	0.0140 (6)	−0.0072 (6)
O6	0.0319 (7)	0.0353 (8)	0.0336 (7)	−0.0122 (5)	0.0171 (6)	−0.0089 (6)
C12	0.0436 (11)	0.0375 (12)	0.0457 (12)	−0.0190 (9)	0.0211 (10)	−0.0029 (10)
C13	0.0349 (11)	0.0456 (13)	0.0403 (11)	−0.0150 (9)	0.0185 (9)	−0.0003 (9)

C14	0.0335 (10)	0.0326 (11)	0.0313 (10)	0.0018 (8)	0.0184 (8)	0.0070 (8)
C15	0.0361 (11)	0.0466 (13)	0.0452 (11)	−0.0001 (9)	0.0233 (9)	0.0085 (10)
C16	0.0453 (12)	0.0474 (13)	0.0497 (12)	0.0135 (10)	0.0329 (11)	0.0152 (10)
C17	0.0574 (14)	0.0332 (12)	0.0423 (11)	0.0107 (10)	0.0317 (11)	0.0088 (9)
C18	0.0425 (11)	0.0332 (11)	0.0346 (10)	0.0025 (9)	0.0199 (9)	0.0040 (9)
C19	0.0334 (10)	0.0309 (10)	0.0291 (9)	0.0035 (8)	0.0169 (8)	0.0084 (8)
C20	0.0347 (10)	0.0451 (13)	0.0372 (11)	−0.0098 (9)	0.0164 (9)	−0.0099 (9)
C21	0.0304 (10)	0.0640 (15)	0.0354 (11)	−0.0074 (10)	0.0133 (9)	−0.0094 (10)
C22	0.0432 (12)	0.0334 (12)	0.0397 (11)	−0.0067 (9)	0.0071 (9)	−0.0085 (9)
C23	0.0307 (10)	0.0394 (12)	0.0400 (11)	−0.0130 (8)	0.0131 (9)	−0.0057 (9)
C24	0.0286 (9)	0.0210 (10)	0.0364 (10)	0.0022 (7)	0.0156 (8)	0.0017 (8)
C25	0.0297 (10)	0.0359 (11)	0.0414 (11)	−0.0034 (8)	0.0160 (9)	0.0017 (9)
C26	0.0400 (11)	0.0441 (13)	0.0484 (12)	0.0020 (9)	0.0278 (10)	0.0097 (10)
C27	0.0468 (12)	0.0364 (12)	0.0361 (11)	0.0047 (9)	0.0235 (10)	0.0037 (9)
C28	0.0362 (10)	0.0257 (10)	0.0349 (10)	0.0013 (8)	0.0160 (8)	−0.0002 (8)
C29	0.0289 (9)	0.0216 (9)	0.0351 (10)	0.0010 (7)	0.0165 (8)	−0.0001 (8)
C30	0.0315 (10)	0.0428 (12)	0.0373 (11)	−0.0145 (9)	0.0143 (9)	−0.0121 (9)
C31	0.0343 (10)	0.0336 (11)	0.0435 (11)	−0.0113 (8)	0.0164 (9)	−0.0108 (9)

Geometric parameters (Å, º)

K—N1	2.8515 (18)	O6—C30	1.443 (2)
K—N2	2.7935 (16)	C12—H12A	0.990
K—O1	2.8156 (13)	C12—H12B	0.990
K—O2	2.8578 (12)	C12—C13	1.495 (3)
K—O3	2.8519 (12)	C13—H13A	0.990
K—O4	2.7138 (13)	C13—H13B	0.990
K—O5	2.8374 (12)	C14—C15	1.386 (3)
K—O6	2.8194 (12)	C14—C19	1.398 (3)
N1—C1	1.331 (3)	C15—H15A	0.950
N1—C5	1.368 (2)	C15—C16	1.392 (3)
N2—C5	1.340 (2)	C16—H16A	0.950
N2—C6	1.384 (2)	C16—C17	1.369 (3)
C1—H1A	0.950	C17—H17A	0.950
C1—C2	1.372 (3)	C17—C18	1.387 (3)
C2—H2A	0.950	C18—H18A	0.950
C2—C3	1.393 (3)	C18—C19	1.380 (3)
C3—H3A	0.950	C20—H20A	0.990
C3—C4	1.364 (3)	C20—H20B	0.990
C4—H4A	0.950	C20—C21	1.506 (3)
C4—C5	1.434 (3)	C21—H21A	0.990
C6—C7	1.408 (3)	C21—H21B	0.990
C6—C11	1.406 (3)	C22—H22A	0.990
C7—H7A	0.950	C22—H22B	0.990
C7—C8	1.373 (3)	C22—C23	1.511 (3)
C8—H8A	0.950	C23—H23A	0.990
C8—C9	1.379 (3)	C23—H23B	0.990
C9—H9A	0.950	C24—C25	1.381 (2)

C9—C10	1.379 (3)	C24—C29	1.407 (2)
C10—H10A	0.950	C25—H25A	0.950
C10—C11	1.380 (3)	C25—C26	1.392 (3)
C11—H11A	0.950	C26—H26A	0.950
O1—C12	1.424 (2)	C26—C27	1.368 (3)
O1—C31	1.427 (2)	C27—H27A	0.950
O2—C13	1.429 (2)	C27—C28	1.391 (3)
O2—C14	1.375 (2)	C28—H28A	0.950
O3—C19	1.387 (2)	C28—C29	1.389 (2)
O3—C20	1.432 (2)	C30—H30A	0.990
O4—C21	1.423 (2)	C30—H30B	0.990
O4—C22	1.395 (2)	C30—C31	1.497 (3)
O5—C23	1.443 (2)	C31—H31A	0.990
O5—C24	1.378 (2)	C31—H31B	0.990
O6—C29	1.372 (2)		

N1—K—N2	47.72 (5)	O1—C12—H12A	109.9
N1—K—O1	108.77 (4)	O1—C12—H12B	109.9
N1—K—O2	90.77 (4)	O1—C12—C13	109.13 (16)
N1—K—O3	90.44 (4)	H12A—C12—H12B	108.3
N1—K—O4	86.90 (5)	H12A—C12—C13	109.9
N1—K—O5	127.32 (4)	H12B—C12—C13	109.9
N1—K—O6	129.29 (4)	O2—C13—C12	108.66 (15)
N2—K—O1	92.68 (4)	O2—C13—H13A	110.0
N2—K—O2	122.03 (4)	O2—C13—H13B	110.0
N2—K—O3	137.39 (5)	C12—C13—H13A	110.0
N2—K—O4	101.98 (5)	C12—C13—H13B	110.0
N2—K—O5	96.91 (4)	H13A—C13—H13B	108.3
N2—K—O6	82.20 (4)	O2—C14—C15	125.02 (17)
O1—K—O2	60.36 (4)	O2—C14—C19	115.82 (15)
O1—K—O3	111.51 (4)	C15—C14—C19	119.16 (18)
O1—K—O4	163.51 (4)	C14—C15—H15A	120.0
O1—K—O5	110.67 (4)	C14—C15—C16	120.02 (19)
O1—K—O6	58.97 (3)	H15A—C15—C16	120.0
O2—K—O3	54.13 (3)	C15—C16—H16A	119.6
O2—K—O4	115.92 (4)	C15—C16—C17	120.71 (18)
O2—K—O5	139.28 (4)	H16A—C16—C17	119.6
O2—K—O6	114.84 (4)	C16—C17—H17A	120.2
O3—K—O4	61.85 (4)	C16—C17—C18	119.5 (2)
O3—K—O5	105.76 (4)	H17A—C17—C18	120.2
O3—K—O6	140.22 (4)	C17—C18—H18A	119.7
O4—K—O5	60.48 (4)	C17—C18—C19	120.58 (19)
O4—K—O6	115.21 (4)	H18A—C18—C19	119.7
O5—K—O6	54.89 (3)	O3—C19—C14	115.69 (16)
K—N1—C1	143.34 (14)	O3—C19—C18	124.32 (17)
K—N1—C5	96.70 (12)	C14—C19—C18	119.99 (17)
C1—N1—C5	118.40 (18)	O3—C20—H20A	110.1
K—N2—C5	100.10 (11)	O3—C20—H20B	110.1

K—N2—C6	136.59 (12)	O3—C20—C21	107.87 (15)
C5—N2—C6	123.11 (16)	H20A—C20—H20B	108.4
N1—C1—H1A	116.8	H20A—C20—C21	110.1
N1—C1—C2	126.4 (2)	H20B—C20—C21	110.1
H1A—C1—C2	116.8	O4—C21—C20	115.52 (17)
C1—C2—H2A	122.0	O4—C21—H21A	108.4
C1—C2—C3	116.1 (2)	O4—C21—H21B	108.4
H2A—C2—C3	122.0	C20—C21—H21A	108.4
C2—C3—H3A	119.9	C20—C21—H21B	108.4
C2—C3—C4	120.1 (2)	H21A—C21—H21B	107.5
H3A—C3—C4	119.9	O4—C22—H22A	109.7
C3—C4—H4A	119.6	O4—C22—H22B	109.7
C3—C4—C5	120.73 (19)	O4—C22—C23	109.93 (16)
H4A—C4—C5	119.6	H22A—C22—H22B	108.2
N1—C5—N2	115.03 (17)	H22A—C22—C23	109.7
N1—C5—C4	118.25 (18)	H22B—C22—C23	109.7
N2—C5—C4	126.56 (17)	O5—C23—C22	107.88 (14)
N2—C6—C7	118.44 (18)	O5—C23—H23A	110.1
N2—C6—C11	125.59 (18)	O5—C23—H23B	110.1
C7—C6—C11	115.73 (18)	C22—C23—H23A	110.1
C6—C7—H7A	118.9	C22—C23—H23B	110.1
C6—C7—C8	122.2 (2)	H23A—C23—H23B	108.4
H7A—C7—C8	118.9	O5—C24—C25	124.30 (16)
C7—C8—H8A	119.6	O5—C24—C29	115.98 (14)
C7—C8—C9	120.8 (2)	C25—C24—C29	119.73 (16)
H8A—C8—C9	119.6	C24—C25—H25A	120.0
C8—C9—H9A	120.7	C24—C25—C26	119.94 (18)
C8—C9—C10	118.5 (2)	H25A—C25—C26	120.0
H9A—C9—C10	120.7	C25—C26—H26A	119.7
C9—C10—H10A	119.4	C25—C26—C27	120.56 (18)
C9—C10—C11	121.1 (2)	H26A—C26—C27	119.7
H10A—C10—C11	119.4	C26—C27—H27A	119.9
C6—C11—C10	121.6 (2)	C26—C27—C28	120.25 (18)
C6—C11—H11A	119.2	H27A—C27—C28	119.9
C10—C11—H11A	119.2	C27—C28—H28A	120.0
K—O1—C12	98.25 (10)	C27—C28—C29	119.95 (18)
K—O1—C31	97.49 (9)	H28A—C28—C29	120.0
C12—O1—C31	111.80 (14)	O6—C29—C24	115.71 (15)
K—O2—C13	113.71 (10)	O6—C29—C28	124.77 (16)
K—O2—C14	125.00 (10)	C24—C29—C28	119.51 (16)
C13—O2—C14	116.73 (14)	O6—C30—H30A	110.0
K—O3—C19	124.46 (10)	O6—C30—H30B	110.0
K—O3—C20	116.65 (10)	O6—C30—C31	108.36 (14)
C19—O3—C20	117.11 (14)	H30A—C30—H30B	108.4
K—O4—C21	110.08 (10)	H30A—C30—C31	110.0
K—O4—C22	112.03 (11)	H30B—C30—C31	110.0
C21—O4—C22	113.97 (16)	O1—C31—C30	109.54 (16)
K—O5—C23	115.88 (10)	O1—C31—H31A	109.8

K—O5—C24	123.80 (10)	O1—C31—H31B	109.8
C23—O5—C24	116.09 (13)	C30—C31—H31A	109.8
K—O6—C29	125.40 (10)	C30—C31—H31B	109.8
K—O6—C30	115.23 (10)	H31A—C31—H31B	108.2
C29—O6—C30	117.00 (13)		

N2—K—N1—C1	−167.6 (2)	N2—K—O4—C21	109.38 (13)
N2—K—N1—C5	−3.90 (9)	N2—K—O4—C22	−122.70 (12)
O1—K—N1—C1	116.4 (2)	O1—K—O4—C21	−98.33 (18)
O1—K—N1—C5	−79.88 (11)	O1—K—O4—C22	29.6 (2)
O2—K—N1—C1	57.6 (2)	O2—K—O4—C21	−25.52 (14)
O2—K—N1—C5	−138.70 (10)	O2—K—O4—C22	102.39 (12)
O3—K—N1—C1	3.4 (2)	O3—K—O4—C21	−28.33 (12)
O3—K—N1—C5	167.17 (10)	O3—K—O4—C22	99.59 (13)
O4—K—N1—C1	−58.4 (2)	O5—K—O4—C21	−159.41 (14)
O4—K—N1—C5	105.39 (10)	O5—K—O4—C22	−31.49 (11)
O5—K—N1—C1	−106.9 (2)	O6—K—O4—C21	−163.69 (12)
O5—K—N1—C5	56.83 (12)	O6—K—O4—C22	−35.77 (13)
O6—K—N1—C1	−178.8 (2)	N1—K—O5—C23	57.94 (13)
O6—K—N1—C5	−15.06 (13)	N1—K—O5—C24	−97.97 (12)
N1—K—N2—C5	4.02 (10)	N2—K—O5—C23	98.49 (12)
N1—K—N2—C6	−170.6 (2)	N2—K—O5—C24	−57.42 (12)
O1—K—N2—C5	117.14 (11)	O1—K—O5—C23	−166.00 (11)
O1—K—N2—C6	−57.47 (18)	O1—K—O5—C24	38.09 (13)
O2—K—N2—C5	60.83 (12)	O2—K—O5—C23	−97.83 (12)
O2—K—N2—C6	−113.78 (17)	O2—K—O5—C24	106.26 (12)
O3—K—N2—C5	−9.24 (14)	O3—K—O5—C23	−45.08 (12)
O3—K—N2—C6	176.14 (15)	O3—K—O5—C24	159.01 (11)
O4—K—N2—C5	−70.45 (11)	O4—K—O5—C23	−1.40 (11)
O4—K—N2—C6	114.94 (18)	O4—K—O5—C24	−157.31 (13)
O5—K—N2—C5	−131.65 (11)	O6—K—O5—C23	173.87 (13)
O5—K—N2—C6	53.73 (18)	O6—K—O5—C24	17.96 (11)
O6—K—N2—C5	175.32 (11)	N1—K—O6—C29	96.36 (13)
O6—K—N2—C6	0.71 (17)	N1—K—O6—C30	−65.53 (13)
K—N1—C1—C2	161.11 (16)	N2—K—O6—C29	88.05 (13)
C5—N1—C1—C2	−0.5 (3)	N2—K—O6—C30	−73.84 (12)
N1—C1—C2—C3	−0.5 (3)	O1—K—O6—C29	−174.04 (14)
C1—C2—C3—C4	1.9 (3)	O1—K—O6—C30	24.06 (11)
C2—C3—C4—C5	−2.5 (3)	O2—K—O6—C29	−150.19 (12)
K—N2—C5—N1	−6.85 (17)	O2—K—O6—C30	47.92 (13)
K—N2—C5—C4	168.44 (16)	O3—K—O6—C29	−87.12 (13)
C6—N2—C5—N1	168.74 (17)	O3—K—O6—C30	110.98 (12)
C6—N2—C5—C4	−16.0 (3)	O4—K—O6—C29	−11.57 (13)
K—N1—C5—N2	6.65 (16)	O4—K—O6—C30	−173.46 (12)
K—N1—C5—C4	−169.05 (14)	O5—K—O6—C29	−16.12 (11)
C1—N1—C5—N2	175.69 (17)	O5—K—O6—C30	−178.01 (13)
C1—N1—C5—C4	0.0 (3)	K—O1—C12—C13	−77.99 (15)
C3—C4—C5—N1	1.5 (3)	C31—O1—C12—C13	−179.51 (15)

C3—C4—C5—N2	−173.69 (18)	K—O2—C13—C12	−17.02 (18)
K—N2—C6—C7	−41.0 (3)	C14—O2—C13—C12	−174.33 (15)
K—N2—C6—C11	133.05 (18)	O1—C12—C13—O2	68.2 (2)
C5—N2—C6—C7	145.31 (18)	K—O2—C14—C15	−163.90 (14)
C5—N2—C6—C11	−40.6 (3)	K—O2—C14—C19	16.4 (2)
N2—C6—C7—C8	175.99 (18)	C13—O2—C14—C15	−9.4 (3)
C11—C6—C7—C8	1.3 (3)	C13—O2—C14—C19	170.87 (15)
C6—C7—C8—C9	−0.8 (3)	O2—C14—C15—C16	178.94 (17)
C7—C8—C9—C10	−0.3 (3)	C19—C14—C15—C16	−1.4 (3)
C8—C9—C10—C11	0.8 (3)	C14—C15—C16—C17	0.1 (3)
C9—C10—C11—C6	−0.2 (3)	C15—C16—C17—C18	0.8 (3)
N2—C6—C11—C10	−175.05 (18)	C16—C17—C18—C19	−0.3 (3)
C7—C6—C11—C10	−0.8 (3)	C17—C18—C19—O3	178.15 (16)
N1—K—O1—C12	−36.51 (11)	C17—C18—C19—C14	−1.0 (3)
N1—K—O1—C31	76.91 (10)	K—O3—C19—C14	−20.1 (2)
N2—K—O1—C12	−82.45 (11)	K—O3—C19—C18	160.64 (13)
N2—K—O1—C31	30.97 (10)	C20—O3—C19—C14	175.63 (15)
O2—K—O1—C12	43.30 (10)	C20—O3—C19—C18	−3.6 (2)
O2—K—O1—C31	156.72 (11)	O2—C14—C19—O3	2.3 (2)
O3—K—O1—C12	61.69 (11)	O2—C14—C19—C18	−178.44 (15)
O3—K—O1—C31	175.11 (10)	C15—C14—C19—O3	−177.40 (16)
O4—K—O1—C12	124.64 (16)	C15—C14—C19—C18	1.9 (3)
O4—K—O1—C31	−121.93 (16)	K—O3—C20—C21	25.82 (19)
O5—K—O1—C12	179.13 (10)	C19—O3—C20—C21	−168.71 (16)
O5—K—O1—C31	−67.44 (10)	K—O4—C21—C20	59.5 (2)
O6—K—O1—C12	−161.68 (12)	C22—O4—C21—C20	−67.4 (2)
O6—K—O1—C31	−48.26 (10)	O3—C20—C21—O4	−58.0 (2)
N1—K—O2—C13	96.99 (12)	K—O4—C22—C23	63.26 (17)
N1—K—O2—C14	−107.89 (13)	C21—O4—C22—C23	−170.92 (14)
N2—K—O2—C13	58.72 (13)	K—O5—C23—C22	30.54 (19)
N2—K—O2—C14	−146.15 (12)	C24—O5—C23—C22	−171.65 (15)
O1—K—O2—C13	−14.27 (11)	O4—C22—C23—O5	−62.1 (2)
O1—K—O2—C14	140.86 (13)	K—O5—C24—C25	160.33 (14)
O3—K—O2—C13	−173.03 (13)	K—O5—C24—C29	−19.7 (2)
O3—K—O2—C14	−17.91 (11)	C23—O5—C24—C25	4.5 (2)
O4—K—O2—C13	−176.09 (12)	C23—O5—C24—C29	−175.53 (16)
O4—K—O2—C14	−20.96 (13)	O5—C24—C25—C26	178.66 (17)
O5—K—O2—C13	−102.07 (13)	C29—C24—C25—C26	−1.3 (3)
O5—K—O2—C14	53.06 (14)	C24—C25—C26—C27	−0.6 (3)
O6—K—O2—C13	−37.77 (13)	C25—C26—C27—C28	1.0 (3)
O6—K—O2—C14	117.36 (12)	C26—C27—C28—C29	0.6 (3)
N1—K—O3—C19	109.73 (13)	K—O6—C29—C24	13.9 (2)
N1—K—O3—C20	−85.99 (12)	K—O6—C29—C28	−165.13 (13)
N2—K—O3—C19	119.50 (13)	C30—O6—C29—C24	175.55 (16)
N2—K—O3—C20	−76.22 (14)	C30—O6—C29—C28	−3.5 (3)
O1—K—O3—C19	−0.69 (13)	C27—C28—C29—O6	176.51 (17)
O1—K—O3—C20	163.59 (12)	C27—C28—C29—C24	−2.5 (3)
O2—K—O3—C19	19.09 (12)	O5—C24—C29—O6	3.8 (2)

O2—K—O3—C20	−176.63 (13)	O5—C24—C29—C28	−177.12 (15)
O4—K—O3—C19	−164.03 (13)	C25—C24—C29—O6	−176.23 (16)
O4—K—O3—C20	0.26 (12)	C25—C24—C29—C28	2.9 (3)
O5—K—O3—C19	−121.06 (12)	K—O6—C30—C31	4.57 (19)
O5—K—O3—C20	43.22 (13)	C29—O6—C30—C31	−158.91 (15)
O6—K—O3—C19	−67.58 (14)	K—O1—C31—C30	78.67 (14)
O6—K—O3—C20	96.71 (13)	C12—O1—C31—C30	−179.30 (15)
N1—K—O4—C21	63.83 (13)	O6—C30—C31—O1	−59.1 (2)
N1—K—O4—C22	−168.25 (12)		